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PROCESS SCALE-UP FOR MERCURIC - 5 - NITROTETRAZOLE

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Scale-up studies were conducted on an improved process for the synthesis of mercuric-5-nitrotetrazole, and a procedure was established for an almost entirely remotely controlled operation. The procedure was demonstrated by the preparation of six one-pound batches and a batch was prepared using well water in the process rather than distilled water so that a comparison could be made of product purity. Process procedures and equipment were suggested for larger scaled production.

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ABSTRACT

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CONTENTS

TABLES

FIGURES

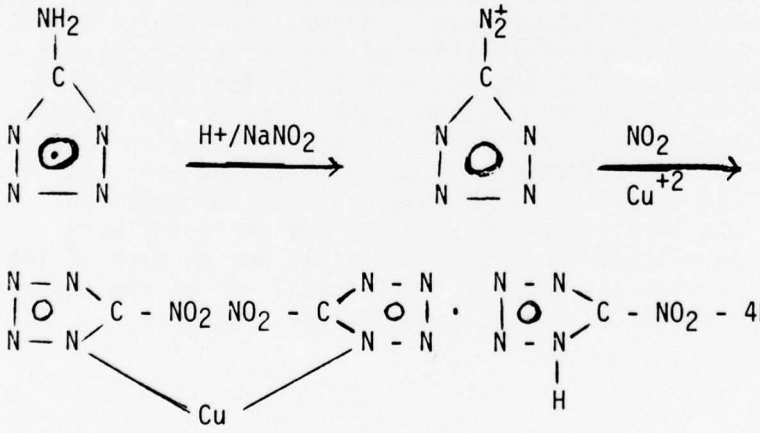
PROCESS SCALE-UP OF MERCURIC-5-NITROTETRAZOLE

INTRODUCTION

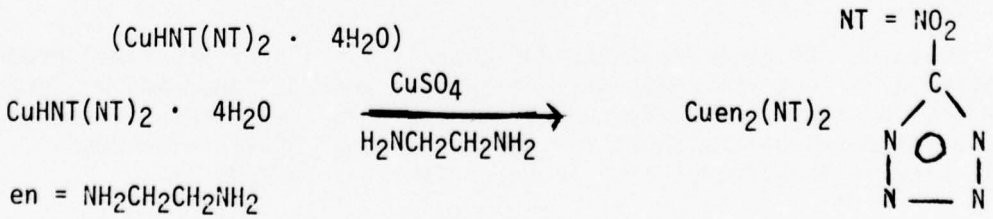
Mercuric-5-nitrotetrazole is a thermally and hydrolytically stable detonant that has been proposed as a replacement for lead azide. Since lead azide is not hydrolytically stable, it can deteriorate on storage through a facile reaction with moisture that is accelerated by carbon dioxide and gaseous emanants from plasticizers and rubbers. The hydrolysis products can react with copper to form copper azide which is extremely shock and thermally sensitive, and a number of accidental explosions have been attributed to lead azide having been stored in copper containing metals. This problem will not occur with mercuric-5-nitrotetrazole.

Because of the potential usefulness of mercuric-5-nitrotetrazole, an improved process was developed for its synthesis at the Naval Surface Weapons Center[1]. The sequence of reactions used in the process are:

1. Preparation of bis(ethylenediamine) copper (II) bis(5-nitrotetrazole) ($Cuen_2(NT)_2$):



$(CuHNT(NT)_2 \cdot 4H_2O)$



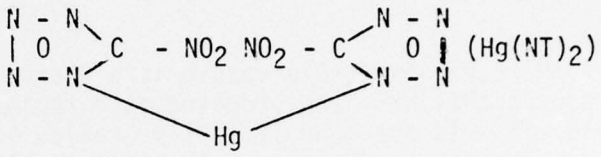
en = $NH_2CH_2CH_2NH_2$

(1) Gilligan, W. and Scott, C., "Mercuric-5-Nitrotetrazole", 1976 Annual Meeting of Pyrotechnics and Explosives, Applications Section, Sunnyvale, CA, November 1976.

2. Conversion of the ethylene diamine complex to mercuric-5-nitro-tetrazole:

$$Cuen_2(NT)_2 + 4HNO_3 \longrightarrow Cu(NT)_2 + en_2 \cdot 4HNO_3$$

$$Cu(NT)_2 + Hg(NO_3)_2 \longrightarrow$$



Scale-up studies of the above synthesis route of mercuric-5-nitro-tetrazole were conducted. Six batches yielding from 65 to 340 grams of the product and six 500-gram batches were prepared. A procedure has been established for an almost entirely remotely controlled operation which can be run routinely. Based on the experience of this work, recommendations have been formulated for further scale-up operations.

DISCUSSION

I. Process Before Scale-Up

The laboratory procedure developed at NSWC, White Oak for the improved synthesis route for mercuric-5-nitrotetrazole consisted entirely of manual operations and was followed by the preparation of the first two batches at the Indian Head laboratories. Filtering and washing of the intermediate $CuHNT(NT)_2 \cdot 4H_2O$ was accomplished on a Buchner funnel requiring manual transfer of both the initial slurry and the sticky,shock-sensitive retentate. Similarly workup of the second intermediate, $Cuen_2(NT)_2$, and the final product were also performed manually. Even though all of the reactions were run behind heavy protective shielding, laboratory personnel were nevertheless exposed to potentially hazardous materials during each of the washing, filtering, and transfering steps. Further scale-up of the process clearly demanded the development of remotely controlled procedures.

II. Scale-up Process

A. General Set-up

Based on the observed handling characteristics of the final product, its intermediates and the reactants, techniques were designed and equipment assembled for an almost entirely remotely controlled operation. Sketches of the apparatus set-up are shown in Figures 1 and 2. Batch sizes and materials used in the process are listed in Tables I through IV.

The reactor was mounted in a water bath in an armor-walled cubicle and almost all of the operations were controlled from a panel on the opposite side of one wall. Visual observations were made through a 1-1/4-inch thick laminated glass window.

B. Liquid Additions

All liquids were added to the reactor through a polyethylene delivery tube extending through the wall to a dropping funnel mounted on the control panel. During the addition of solution B, solids occasionaly were deposited at the tip of the tube but they could easily be rinsed off with a little water. If solution B was not maintained at about 40°C during the addition, a small amount of solids occasionally precipitated in the dropping funnel, slowing the fluid flow.

C. Agitation

An air driven stirring motor was controlled by a valve at the control panel. Smooth and efficient stirring is necessary to ensure complete reactions and aid in adequate temperature control. Without adequate agitation during the addition of solution B, $CuHNT(NT)_2 \cdot 4H_2O$ tends to collect on the surface of the reaction mixture, preventing even distribution of the reagent and hindering control of the reaction.

D. Temperature Control

The reactor was mounted in a bath filled with either ice cooled or steam heated water, and the steam line was controlled by a thermostatically operated valve. This set-up provided adequate temperature control for small batches run in glass beakers; however, it did not provide sufficient heat transfer to cope with the larger batches using less thermally conductive, heavy-walled polypropylene reactors.

Starting with the preparation of Batch #9 of the copper complexes, the heat transfer was increased by two techniques. The water bath was agitated by bubbling air through it, and hot or cold water was pumped through a coil of stainless steel tubing immersed in the reactor. In this way, temperatures from below 5°C to above 75°C could be quickly achieved and easily maintained. As a result, approximately 40% less time was required for the addition of solution B to A where the reaction temperature must be maintained below 18°C.

In Batch #8, the immersion coil was not used, adequate temperature control was difficult, and the final product which is Batch #6 of mercuric-5-nitrotetrazole, had a purity of only 89%. During the preparation of $Cuen_2(NT)_2$, a temperature of only 50°C could be attained, and during its subsequent conversion to mercuric-5-nitrotetrazole, a temperature of only 65°C could be reached. To ensure complete reactions, both steps should have been run at 75°C. During the crystallization of $Cuen_2(NT)_2$, the poor heat transfer resulted in very slow cooling. The dark blue product was obtained in the form of unusually fine crystals which were difficult to wash thoroughly and which remained contaminated with some of the green $CuHNT(NT)_2 \cdot 4H_2O$.

In each step of the process, temperature control is essential. Even after the final step, the preparation of mercuric-5-nitrotetrazole, the slurry must be cooled from 75°C to 55°C before filtering. Insufficient cooling may result in product loss, while cooling below 55°C will precipitate other salts, contaminating the product and lowering the purity. However, during this final process, the immersion coil was not used because of potential safety problems. The final product if spattered on the hot coil might have evaporated to dryness or might have been difficult to remove. Necessary heat was obtained by using preheated water.

Further scale-up studies should be run in stainless steel equipment. This would improve the heat transfer and probably eliminate the need for an immersion coil.

E. Filtration

Filtering was accomplished with an immersion filter disk attached to a bar extending from a remotely controlled power laboratory jack. When not in use, the disk was suspended above the reactor, and during the first step in the process, it served as a vacuum exhaust system for the removal of substantial amounts of NO_2 fumes which are generated as a by-product. (During the preparation of Batches #3 and #4 of the $CuHNT(NT)_2 \cdot 4H_2O$ reactions, the NO_2 fumes caused some deterioration of rubber connections used in the set-up, resulting in a few black particles contaminating the product). When filtration was necessary, the porous disk was easily immersed into the reaction solution by lowering the power laboratory jack, and the filtrate was collected in a heavy-walled flask on the opposite side of the wall next to the control panel. The flask was equipped with a vacuum gauge and connected to the filter disk by polyethylene tubing.

Frequently during the filtering and washing of the first intermediate, $CuHNT(NT)_2 \cdot 4H_2O$, the porous disk became excessively packed with the sticky solid, and filtration ceased. At this point, the filter disk was raised, the filtrate collection flask was disconnected, and water or air was pumped back through the polyethylene tubing, forcing the filter-cake to drop.

Filtration steps, particularly of the $CuHNT(NT)_2 \cdot 4H_2O$, were the most troublesome and time-consuming parts of the process. Generally, a filter disk having 35 micron pores was used, which effectively retained the solids but usually permitted only slow filtration and required frequent back-flushing to cope with clogging. An attempt was made to alleviate the problem by using a disk having 70 micron pores but it was too coarse to retain the $CuHNT(NT)_2 \cdot 4H_2O$, and 20% of Batch #7 was sucked through the filter disk and lost.

Because of the filtration problems, the filtering and washing sequence of $CuHNT(NT)_2 \cdot 4H_2O$ often required about six hours for completion.

For convenience the operation could have been interrupted by the addition of extra water and completed on the following day, but in practice the process was always continued through the preparation of the second intermediate, $Cuen_2(NT)_2$.

The ethylene diamine complex, which is only slightly shock sensitive, was transferred to a fume hood and filtered and washed manually on a Buchner funnel behind a shield. It was then dried overnight by sucking air through the funnel, and the residual water content was usually less than 0.1%.

F. Rinsing of Reactor

Before and after each step of the process, efficient rinsing of the reactor walls, the stirring shaft, the immersion coil, and the filter is recommended. This is particularly important since a vigorous agitation spatters droplets of the reaction mixture which evaporate and leave a solid deposit. Inefficient rinsing of spattered solids may leave unreacted material to contaminate the product.

In practice the rinsing operation was performed manually with a squirt bottle and was not always effective. As this was particularly true for deposits of $Cu(NT)_2$, hot water was added to raise the level of the reaction mixture above the splash zone. It could have been accomplished remotely with a high pressure shower nozzle positioned above the reactor.

G. Product Transfer

After being thoroughly washed, the shock-sensitive final product was transferred as an aqueous slurry to plastic shipping containers. The transfer was accomplished safely and efficiently using the suction device shown in Figure 2, thus avoiding manual scooping or pouring of the slurry. The final yield of product could only be estimated because drying was prohibited and its density was unknown. The product slurry was shipped to NSWC, White Oak, Maryland, for analysis and evaluation.

H. Use of Well Water

Because of the large volume of water required in the process, the use of distilled water would add greatly to the cost of large-scale production. Moreover, the process would be substantially simplified if well water could be used to produce a product having adequate purity.

After completion of the requested scale-up studies using only distilled water, an additional one-pound batch, Batch #12, was made using ordinary well water. A typical analysis for the metal content in the water is given in Table V. The product was shipped to NSWC, White Oak, for analysis and for a comparison of its purity and characteristic with material prepared using distilled water. The product was found to be 100.6% pure and it appears that well water can be used satisfactorily in further process scale-up, simplifying the manufacture and reducing the cost of mercuric-5-nitrotetrazole.

I. Recommendations for Future Scale-up

For larger scale production of $CuHNT(NT)_2 \cdot 4H_2O$, the proposed reactor is a jacketed,agitated,open top container(See Figure 3). The size of this reactor should be at least 12 liters per kg of copper salt. After the reaction is complete, the $CuHNT(NT)_2 \cdot 4H_2O$ slurry should be filtered in a plate and frame filter having a size of 1.0 cm^2/gram of solids. The 10% nitric acid and water washes should be sprayed into the reactor to wash the product into the filter. The $CuHNT(NT)_2 \cdot 4H_2O$ collected in the filter would be dissolved in 80°C aqueous solution of ethylenediamine and cupric sulfate, and pumped into a $Cuen_2(NT)_2$ crystallizer.The crystallizer size should be 9 liter/kg of product. This solution should be cooled to 0 to 5°C and filtered into a Nutsche filter.

The proposed reactor for $Hg(NT)_2$ preparation is a jacketed, agitated, open top container. The size of which should be at least 17 liters per kg of $Hg(NT)_2$. The reaction and wash solutions should be removed by an immersion filter, and the product transferred into shipping containers in equipment similar to that shown in Figure 2.

The conversion of the dissolved $Cuen_2(NT)_2$ to a slurry of insoluble $Cu(NT)_2$ is achieved by the addition of nitric acid. For monitoring this reaction, it is proposed to use a cupric ion specific electrode. The operational range for this electrode is as follows:

Concentration of Cu^{++} -	1.0 to 10^{-7} molar
pH -	0 to 14
Temperature -	0 to 80°C

The use of the above electrode would also simplify the preparation of $Cuen_2(NT)_2$, since then it would not be necessary to isolate and assay this intermediate. A mercuric ion specific electrode could be used to monitor the last step in the reaction sequence.

EXPERIMENTAL

I. General

Quantities of all reactants used for the preparation of the intermediate $Cuen_2(NT)_2$ are listed in Table I, and those used for the subsequent conversion to mercuric-5-nitrotetrazole are listed in Table III. The types of reactors used, reaction conditions, yields, and observations are summarized in Tables II and IV for the preparation of $Cuen_2(NT)_2$ and mercuric-5-nitrotetrazole, respectively.

II. Preparation of Solution A

The use of $CuSO_4 \cdot 5H_2O$ is recommended for the preparation of Solution A, an aqueous solution of $CuSO_4$ and $NaNO_2$. An equivalent amount of anhydrous $CuSO_4$ may be used but will liberate NO_2 fumes if added to aqueous

$NaNO_2$. To avoid the NO_2 fumes if anhydrous $CuSO_4$ is used, it should be dissolved in water before mixing with $NaNO_2$.

III. General Procedure for Batches No. 1 and No. 2

Solution A was cooled in a 2 liter glass beaker to 5°C and solution B was added dropwise over a period of 90 minutes with efficient stirring and cooling to maintain the temperature at 15 - 18°C. The mixture was stirred for an additional 15 minutes, then solution C was added dropwise and stirring continued for another 30 minutes. The resulting slurry was suction filtered on a Buchner funnel, and the retentate was washed with 1.8 N HNO_3 and water but not allowed to dry druing the process.

The wet filtercake consisting of $CuHNT(NT)_2 \cdot 4H_2O$, was transferred to a 1500 ml Erlenmeyer flask, and enough water was added to make a total volume of 600 ml. While being efficiently stirred, the slurry was heated to 75°C by a hot water bath, and solution D was added. The reaction fluid was stirred until homogeneous,then quickly cooled by agitating in an ice bath. The intermediate, $Cuen_2(NT)_2$, was crystallized and after an additional 45 minutes of cooling, it was collected on a Buchner funnel, washed with cold water, and air-dried. A small portion of the crystals were vacuum-dried to determine their water content prior to the next step.

Solution E was stirred efficiently and heated to 75 - 80°C, to ensure complete dissolving of the $Cuen_2(NT)_2$. Solution F was added dropwise over about 15 minutes and insoluble $Cu(NT)_2$ precipitated in Batch #2 but not in Batch #1 where less nitric acid was used. In both batches, stirring and heating at 75°C were continued and solution G added. In Batch #1, an additional 20 ml of 70% HNO_3 was then added, and solids immediately precipitated. The batches were then cooled to 55°C and the supernatent liquid decanted. The solid product was washed five times by adding and decanting 500 ml portions of water,transferred as a slurry to a plastic container by washing it out with a stream of water, and stored under water.

The crystal form of the mercuric-5-nitrotetrazole prepared in Batch #1 was different from that of subsequent batches; however, its purity was 97%. Safety data on the final product from Batch #1 and on each of the intermediates are summarized in Table VI.

IV. General Procudure for Batches #9 to #15 of $Cuen_2(NT)_2$

Remotely controlled equipment was set up as shown in Figure 1. A three-gallon polypropylene beaker was used as the reactor. Quantities of reagents, reaction conditions, and results are given in Tables I and II for each batch.

The reactor containing Solution A was mounted in an ice-water bath as shown in Figure 1 and cooled to 5°C. Efficient temperature control was achieved by bubbling air through the ice-water, pumping cold water through a coil of stainless steel tubing immersed in the reactor, and vigorously agitating the reaction mixture with an air-operated stirrer. From a reservoir

on the opposite side of a steel plated wall, Solution B at 40°C was added dropwise over two to three hours through a polyethylene delivery tube while the reaction temperature was maintained below 18°C. The NO_2 fumes which were evolved were removed through the suction filter suspended above the reactor. Fifteen minutes after the addition was completed, Solution C was added. After an additional 30 minutes, the agitator was stopped, and the suction filter remotely lowered gradually into the slurry. A 9 cm diameter flat-face immersion filter was used. After 1-2 liters of filtrate had been collected, the vacuum was released. The filter was then raised and back-flushed with air or water to remove the 2-10 mm thick filter cake. This process was repeated until the filtration was complete. The retentate was washed once with 10% nitric acid and five times with water. Before each wash, the filter was raised to minimize clogging while the agitator was turned on. After each wash, it was gradually lowered again and suction applied. The filtering and washing took five to six hours. After the final wash, the filter was raised, the agitator turned on, sufficient water added to make the total volume about 4.5 liters, and hot water was circulated through the bath and the immersion coil. When the temperature of the slurry reached 75°C, Solution D also at 75°C was added. The light green deposits of $CuHNT(NT)_2 \cdot 4H_2O$ on the reactor walls were washed down, and stirring was continued until the mixture was homogenous. By subsequently adding ice to the water bath and circulating cold water through the reactor's immersion coil, the mixture was cooled to below 5°C in about 30 minutes to promote crystallization of $Cuen_2(NT)_2$. The cold slurry was filtered in a 24 centimeter diameter polypropylene Buchner funnel, and the product was washed with 1.4 liters of cold water twice. After air-drying, a 10-gram portion of the crystals was vacuum-dried to determine their water content which was usually found to be less than 0.1%.

V. General Procedure for Batches No. 7 to No. 12 of Mercuric-5-Nitrotetrazole

Equipment was set up as shown in Figure 1, except that the immersion coil was not used in the reactor. Solution E was prepared in the reactor by adding 80°C water to $Cuen_2(NT)_2$. Solution F was added over 30 minutes. When a green slurry was not obtained, approximately 10% additional nitric acid was added. Solution G was added over about one hour, and two liters of 75°C water were added to dissolve the unreacted $Cu(NT)_2$ splashed on the reactor walls. The $Hg(NT)_2$ slurry was cooled to 55°C and filtered with a 6 centimeter diameter flat faced polypropylene immersion filter having 35 micron pores. The product was washed five times with water in less than one hour. Each wash was two liters and was agitated before being filtered. Three liters of water was then added and the $Hg(NT)_2$ was hydraulicly transferred to plastic shipping containers using the device illustrated in Figure 2. Because drying of the product was prohibited and its density was unknown, yields of mercuric-5-nitrotetrazole were conservatively estimated at 80% as based on the intermediate $Cuen_2(NT)_2$.

VI. Waste Treatment

All of the waste solutions were treated with 20% NaOH solution, and the supernatant liquid decanted and incinerated. In larger scale operations the mercury in the solid residue could be recovered. Disposal of the small scale heavy metal waste solids awaits an executive decision.

Table I

MATERIALS USED IN THE PREPARATION OF $Cu(en)_2(NT)_2$

Liquid Added to Reactor	Batch No. 1 - 4		Batch No. 5		Batch No. 6		Batch No. 7 - 9		Batch No. 10 - 15	
Solution A										
Water, ml	300	-	600	-	1200	-	1500	-	2100	-
$NaNO_2$, g (moles)	104	(1.5)	208	(3.0)	416	(6.0)	520	(7.5)	728	(10.5)
$CuSO_4 \cdot 5H_2O$,g(moles)	55	(0.22)	110	(0.44)	220	(0.88)	275	(1.1)	385	(1.54)
Solution B										
Water, ml	600	-	1200	-	2400	-	3000	-	4200	-
70% HNO_3, ml(moles)	64	(1.0)	128	(2.0)	256	(4.0)	320	(5.0)	448	(7)
5-Aminotetrazole $\cdot H_2O$,g(moles)	51.5	(0.5)	103	(1.0)	206	(2.0)	257.5	(2.5)	360.5	(3.5)
$CuSO_4 \cdot 5H_2O$, g(moles)	2	(0.01)	4	(0.02)	8	(0.03)	10	(0.04)	14	(0.06)
Solution C										
Water, ml	30	-	60	-	120	-	150	-	210	-
70% HNO_3, ml(moles)	70	(1.1)	140	(2.2)	280	(4.3)	350	(5.4)	490	(7.5)
Acid Wash										
10% HNO_3, ml	250	-	500	-	1000	-	1300	-	1750	-
Water Wash(5 times)										
Water, ml	250	-	500	-	1000	-	1300	-	1750	-
Total Reaction Solution Volume (before addition of Solution D), ml	600	-	1200	-	2400	-	3000	-	4500	-
Solution D										
Water, ml	80	-	160	-	320	-	400	-	560	-
$CuSO_4 \cdot 5H_2O$,g(moles)	21	(0.08)	42	(0.17)	84	(0.34)	105	(0.42)	147	(1.6)
Ethylenediamine,ml (moles)	45	(0.67)	90	(1.34)	180	(2.7)	225	(3.37)	315	(4.7)
Water Wash (2 times)										
Water, ml	200	-	400	-	800	-	1000	-	1400	-

Table II

SUMMARY OF $Cuen_2(NT)_2$ REACTIONS

Batch No.	5-Amino-tetrazole (moles)	Max. Temp. During Addition of Soln. B (°C)	Addition Time (min.)	Temp. Cooled Before Filtering (°C)	Yield of $Cuen_2(NT)_2$ (%)	(g)	Reactor Type	Remarks
1	0.5	18	45	5	66	68.6	2 l. glass beaker	Batch run in hood, non-remote
2	0.5	18	60	5	63	65.2	2 l. glass beaker	Batch run in hood, non-remote
3	0.5	10	95	2	63	64.5	2 l. glass beaker	NO_2 by-product decomposed rubber adaptors
4	0.5	14	50	8	54	57.5	2 l. glass beaker	NO_2 by-product decomposed rubber adaptors
5	1.0	15	110	3	63	130.5	3 l. glass beaker	
6	2.0	26	235	8	47	193.5	3-1/2 gallon poly-carbonate beaker	Very poor heat transfer, 26°C temperature for only 10 min. rest of react. run at 15°C
7	2.5	11	240	2	46	236.9	3-gallon polypropy-lene beaker	70 micron filter used, cooled in refrigerator from 20 to 2°C
8	2.5	10	253	2	66	340	3-gallon polypro-pylene beaker	Ethylene diamine added at 50°C, cooled in refrig. from 50 to 2°C, very fine crystals of $Cuen_2(NT)_2$ obtained & about 10% of $CuHNT(NT)_2 \cdot 4H_2O$ remained unreacted.
9	2.5	12	105	3	66	339	3-gallon polypro-pylene beaker	Stainless steel coil used reduced heating & cooling times by over 40%
10	3.5	6	157	5	68	492.5	3-gallon polypro-pylene beaker	
11	3.5	6	212	4	71	510.7	3-gallon polypro-pylene beaker	
12	3.5	8	140	4	71	509.9	3-gallon polypro-pylene beaker	
13	3.5	8	-	-	-	-	3-gallon polypro-pylene beaker	Thermometer broke, batch dis-carded
14	3.5	10	150	3	78	562.7	3-gallon polypro-pylene beaker	
15	3.5	8	130	5	64	458	3-gallon polypro-pylene beaker	Well water used, some $Cuen_2(NT)_2$ lost through hole in filter paper.

Table III

MATERIALS USED IN THE PREPARATION OF $Hg(NT)_2$

Batch No.	Solution E			Solution F			Extra	Solution G		Water Wash
	$Cuen(NT)_2$ (g)	$Cuen(NT)_2$ (moles)	Water (ml)	Water (ml)	70% HNO_3 (ml)	70% HNO_3 (moles)	70% HNO_3 (ml)	1 M $Hg(NO_3)_2$ (ml)	70% HNO_3 (ml)	5 times (ml)
1	68.6	0.17	770	110	46	0.7	-	179	4	500
2	65.0	0.16	720	105	43	0.66	5	166	4	500
3	119.5	0.29	1300	195	78	1.2	10	306	5	600
4	132.9	0.32	1460	208	87	1.3	10	341	6	700
5	193.5	0.47	2140	310	126	1.9	13	495	8	1000
6	340	0.83	3800	560	230	3.5	23	870	14	1700
7	500	1.21	5500	800	325	5.0	0	1275	22	2000
8	500	1.21	5500	800	325	5.0	33	1275	22	2000
9	500	1.21	5500	800	325	5.0	33	1275	22	2000
10	500	1.21	5500	800	325	5.0	0	1275	22	2000
11	500	1.21	5500	800	325	5.0	0	1275	22	2000
12	607.5	1.48	6680	975	406	6.2	0	1550	25	3000

Table IV

SUMMARY OF $Hg(NT)_2$ REACTIONS

Batch No.	$Cuen_2(NT)_2$ (g)	$Cuen_2(NT)_2$ Batch No.	Assumed(1) $Hg(NT)_2$ Yield (g)	Purity $Hg(NT)_2$(2) (%)	Reactor Type	Remarks
1	68.6	1	57	97.2±0.6	2 l. glass flask	Modified crystals (20 ml 70% HNO_3 added to crystallize $Hg(NT)_2$ at end of batch)
2	65	2	54	98.2±1.3	2 l. glass flask	
3	119.5	3,4	100	98.8±0.5	4 l. glass beaker	Black pieces of rubber in product from $Cuen_2(NT)_2$ batches
4	132.9	5	110	98.8±1.5	4 l. glass beaker	
5	193.5	6	161	99.0±0.6	4 l. glass beaker	
6	340	8	283	89.3±0.9	3 gal. polypropylene beaker	$Hg(NO_3)_2$ added at 65-69°C; $Hg(NT)_2$ appeared less dense than from other batches
7	500	7,9	400	99.4±0.8	3 gal. polypropylene beaker	30°C distilled water added to heat reaction solution
8	500	10	400	99.4±0.9	3 gal. polypropylene beaker	
9	500	11	400	100.3	3 gal. polypropylene beaker	Product had a light tan color
10	500	12	400	100	3 gal. polypropylene beaker	Product had a light tan color
11	500	14	400	100	3 gal. polypropylene beaker	Product was white in appearance
12	607.5	15	500	100.6	3 gal. polypropylene beaker	Product had a very light tan color; Well water used in place of distilled water

(1) The yield of $Hg(NT)_2$ was assumed to be 80% of the theory.
(2) Purity based on UV absorption at 257μ for nitrotetrazole.

Table V

METAL IMPURITIES IN A TYPICAL SAMPLE OF WELL WATER

Metal	PPM
Cd	0.002
Ca	0.1
Cr	0.02
Cu	0.03
Fe	0.15
Mn	0.01
Mg	0.2
Ag	0.003
Na	30.0
Pb	<0.02
Ti	.004
K	2.0
Sr	0.0006
Al	0.04
Si	6.0
B	0.6

Table VI

SAFETY DATA OF MERCURIC-5-NITROTETRAZOLE [$Hg(NT)_2$] AND ITS INTERMEDIATES[d]

Material	Impact with 5Kg Weight[a] (mm)	Sliding Friction[b] (lbs)	Electrostatic Discharge[c] (joules)
$CuHNT(NT)_2 \cdot 4H_2O$, water wet	≥600 (low relat. sensitivity)	≥980 (low relat. sensitivity)	≥12.5 (low relat. sensitivity)
$Cu\ HNT(NT)_2 \cdot 4H_2O$, dry	50 (high relat. sensitivity)	<40 (high relat. sensitivity)	≥12.5 (low relat. sensitivity)
$Cuen_2\ (NT)_2$, dry	200 (medium relat. sensitivity)	≥980 (low relat. sensitivity)	≥12.5 (low relat. sensitivity)
$Hg(NT)_2$, water wet	50 (high relat. sensitivity)	<40 (high relat. sensitivity)	≥12.5 (low relat. sensitivity)
$Hg(NT)_2$, dry	50 (high relat. sensitivity)	<40 (high relat. sensitivity)	≥12.5 (low relat. sensitivity)

[a] Three consecutive positive tests
[b] 8 ft/sec. 20 consecutive failures
[c] 5000 volts, 20 consecutive failures
[d] Safety tests performed by NAVORDSTA Safety Department

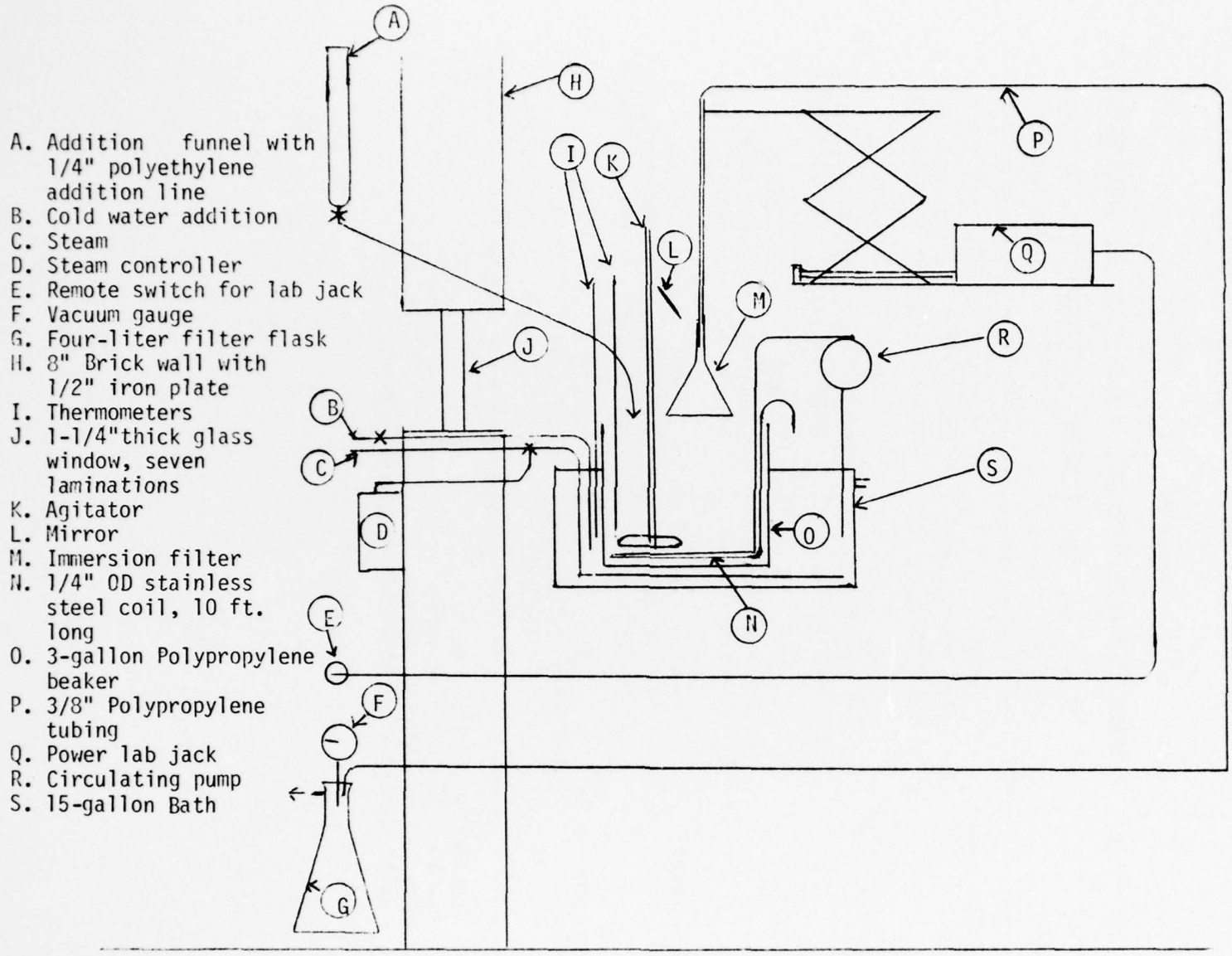


Figure 1: EQUIPMENT FOR PREPARING $Cuen_2$ $(NT)_2$ AND $Hg(NT)_2$

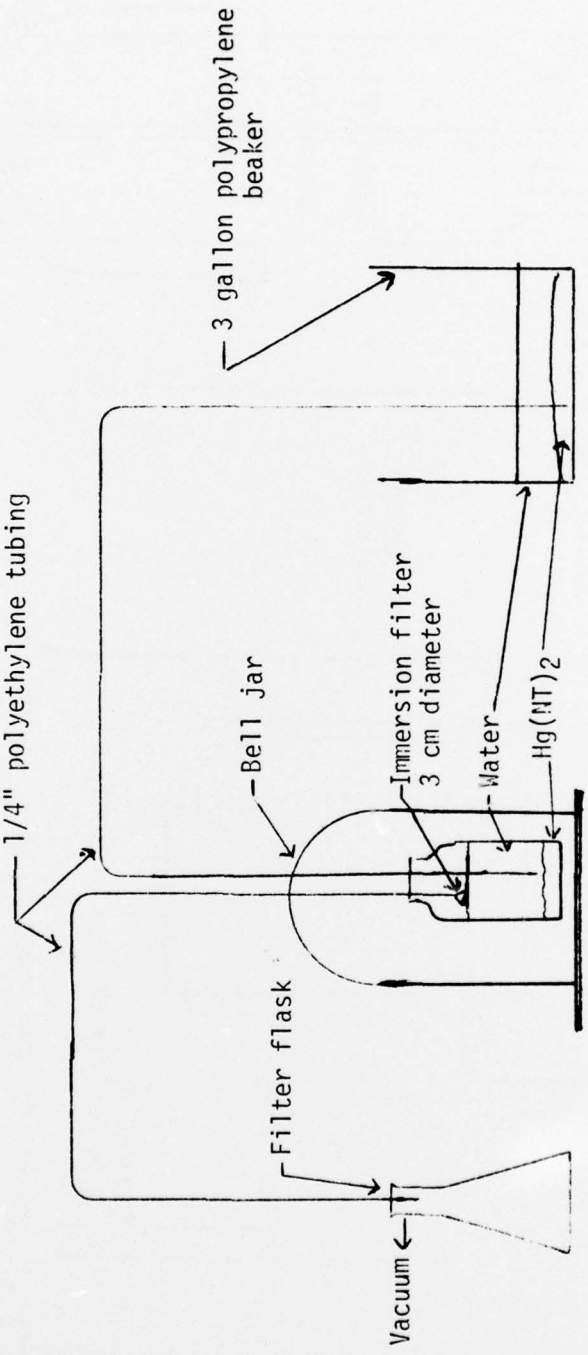


Figure 2: HYDRAULIC TRANSFER OF $Hg(NT)_2$

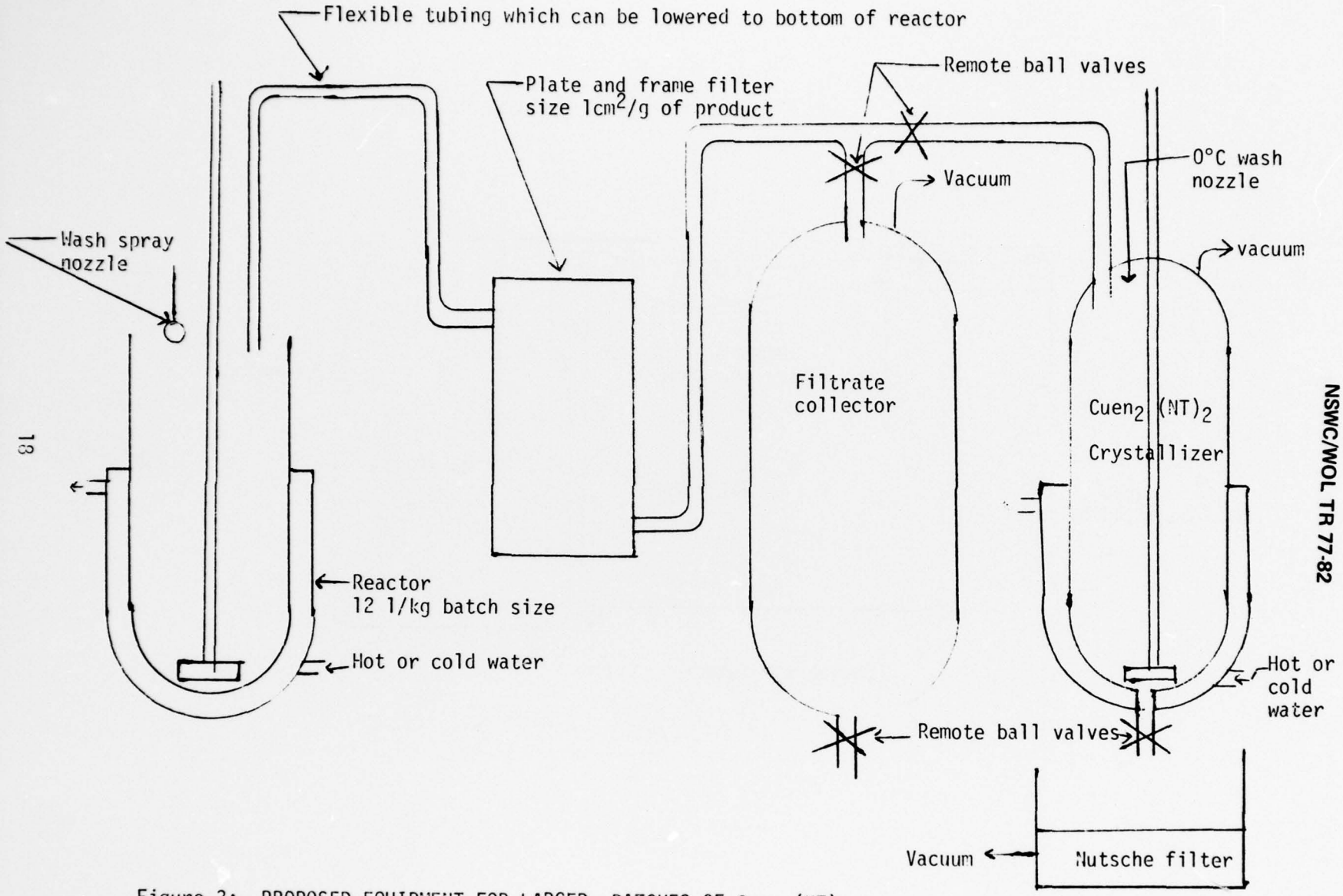


Figure 3: PROPOSED EQUIPMENT FOR LARGER BATCHES OF $Cuen_2(NT)_2$

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